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IS 11153 (1996): Manganese dioxide for dry batteries [CHD 1: Inorganic Chemicals]

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शुष्क बैटरियों के लिए मैंगनीज डाईआक्साइड — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

MANGANESE DIOXIDE FOR DRY BATTERIES — SPECIFICATION

(First Revision)

ICS 71.060.20 : 29.220.10

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

August 1996 **Price Group 7**

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the General Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Manganese dioxide occurs in nature as pyrolusite, ramsdellite, psilomelane, cryptomelane, hollandite, etc. Out of these pyrolusite, ramsdellite, psilomelane are the most important ores of manganese dioxide. India has the fourth largest manganese ore deposits in the world. In order to regulate the export and to develop the quality of indigenous material, the following Indian Standards have already been brought out:

IS 1449 : 1976	Methods of sampling manganese ore (*first revision*)
IS 1473 : 1960	Methods of chemical analysis of manganese ore
IS 4763 : 1982	Specification for manganese ore for production of ferromanganese (*first revision*)
IS 5713 : 1981	Specification for manganese dioxide for explosive and pyrotechnic composition (*first revision*)

Manganese ore is usually associated with other mineral constituents like iron oxide, silica, alumina, phosphorus, sulphur and other heavy metals in traces. For use in the dry leclanche cells, usually high percentage manganese dioxide with particular crystal modification is used and it should be largely free from compounds of metals electropositive to zinc, such as copper, nickel, cobalt, arsenic and antimony.

Effectiveness of manganese dioxide as depolariser in dry cell depends upon its crystal structure. Gamma type crystal structure is most desirable whereas alpha and beta type of manganese dioxide are less effective as depolarisers.

Some naturally occurring battery grade imported manganese dioxide ores contain mineral ramsdellite in which gamma Manganese dioxide is present. Synthetically manufactured products like electrolytic manganese dioxide, chemical manganese dioxide, or activated manganese dioxide also contain manganese dioxide in gamma form. Manganese dioxide ore deposits found in India generally contain beta or alpha type or both.

The physical characteristics of manganese dioxide such as X-ray crystallographic structure, fineness, surface area, etc, are important in the selection of manganese dioxide.

It has been established that the gamma modification of manganese dioxide is the most reactive constituent in the depolariser in dry cell. X-ray diffraction examination is essential to identify the gamma variety. The alpha and delta varieties are also found to be more reactive more than the beta variety.

This standard was originally published in 1984. In this revision a new requirement for particle size has been incorporated along with the relevant test method. Atomic absorption spectrophotometric methods for the determination of lead, copper, nickel and iron have been incorporated as alternate methods. The requirement for activity index for Type B and Type C material has been deleted.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MANGANESE DIOXIDE FOR DRY BATTERIES — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for manganese dioxide suitable for dry batteries.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
1070 : 1992	Reagent grade water (*third revision*)
4905 : 1968	Methods for random sampling

3 TYPES

3.1 The material shall be of the following three types:

Type A — Natural ore,

Type B — Chemically activated manganese dioxide (CMD), and

Type C — Electrolytic manganese dioxide (EMD).

4 REQUIREMENTS

4.1 Description

The material shall be free flowing and in a powdered form.

4.2 Particle Size of the Material

The particle size of the material for Type 'A' shall be as agreed to between the purchaser and the supplier.

4.3 The material, when tested according to the methods prescribed in Annex A, shall comply with the requirements given in Table 1. Reference to the relevant clauses of Annex A is given in col 6 of the table.

Table 1 Requirements for Manganese Dioxide for Dry Batteries

Sl No.	Characteristic	Requirement			Method of Test (Ref to Cl No. in Annex A)
		Type A	Type B	Type C	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Manganese dioxide, percent by mass (on dry basis), *Min*	72	85	90	A-2
ii)	Moisture, percent by mass, *Max*	3	3	3	A-3
iii)	Lead (as Pb), percent by mass, *Max*	0.1	0.1	0.1	A-4[1] or A-14 or A-16
iv)	Iron (as Fe), percent by mass, *Max*	7.0	0.1	0.1	A-5[1] or A-13 or A-16
v)	Sulphate (as SO_4), percent by mass, *Max*	—	1.5	1.5	A-6
vi)	Matter insoluble in hydrochloric acid on ignition, percent by mass, *Max*	10.0	0.5	0.5	A-7
vii)	Copper (as Cu), percent by mass, *Max*	0.02	0.002	0.002	A-8[1] or A-15 or A-16
viii)	Nickel (as Ni), percent by mass, *Max*	0.04	0.003	0.003	A-9 or A-16
ix)	Particle size, passing through 75 micron IS Sieve, percent by mass, *Min*	—	85	85	A-10
x)	*p*H of 5 percent water extract	—	4.5-6.5	4.5-6.5	A-11
xi)	Crystal structure	—	Gamma variety	Gamma variety	A-12

[1] In case of dispute, the methods given in A-4, A-5 and A-8 shall be the referee methods.

5 PACKING AND MARKING

5.1 The material shall be packed in HDPE woven bags with a polyethylene liner inside.

5.2 Marking

Each package shall bear legibly and indelibly the following information:

a) Name and type of the material;
b) Indication of the source of manufacture;
c) Gross and net mass; and
d) Lot number.

5.2.1 The product may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

The method of drawing representative samples from a lot, number of tests to be conducted and the criteria for conformity shall be as prescribed in Annex B.

ANNEX A

(*Clause* 4.3)

METHODS OF TEST FOR MANGANESE DIOXIDE FOR DRY BATTERIES

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF MANGANESE DIOXIDE CONTENT (OXIDATION VALUE)

A-2.0 Principle

Manganese dioxide is treated with known excess quantity of oxalic acid in presence of sulphuric acid, excess oxalic acid is found out by titration with standard potassium permanganate solution and amount of manganese dioxide percent is found out from oxalic acid consumed.

$$MnO_2 + (COOH)_2 + H_2SO_4 - MnSO_4 + 2CO_2 + 2H_2O$$

A-2.1 Reagents

A-2.1.1 *Standard Oxalic Acid Solution* — 0.5 N.

A-2.1.2 *Standard Potassium Permanganate Solution* — 0.1 N (freshly filtered and standardized).

A-2.1.3 *Dilute Sulphuric Acid Solution* — 50 percent.

A-2.1.4 *Sodium Oxalate* — A.R. grade.

A-2.2 Procedure

Weigh about 0.5 g sample accurately (previously ground to pass through 150 micron IS sieve) and transfer to a 500 ml conical flask.

Add 40 ml of dilute sulphuric acid and 100 ml of oxalic acid by pipette. Digest on hot water bath with occasional swirling till all the manganese dioxide particles are decomposed.

Titrate with standard potassium permanganate maintaining temperature at around 70°C till permanent faint pink colour persists (Reading B).

Alternatively, 2 to 3 drops of 1:10 phenanthroline (0.025 M) indicator solution can be used. The end point will be from pink to green.

Find out blank reading for the reagents used by titrating with the standard permanganate solution (Reading A).

A-2.3 Calculations

Manganese dioxide (dry basis), percent by mass

$$= \frac{(A - B) \times N \times 0.043\,47 \times 10\,000}{(100 - W) \times M}$$

where

A = volume in ml of the standard permanganate solution required for the blank,
B = volume in ml of the standard permanganate solution required for the back titration of excess of oxalic acid,
N = normality of the standard permanganate solution,
M = mass in g of the material taken for the test, and
W = moisture in the sample as determined in A-3.

A-3 DETERMINATION OF MOISTURE

A-3.1 Procedure

Weigh accurately about 10 g of the material in a flat bottom petri dish. Place the dish in an electric oven maintained at 120 + 5°C for about 16 hours. Cover the dish, cool in a desiccator and weigh. Repeat the procedure of heating, cooling and weighing at half-hour intervals until the difference between two consecutive weighings is less than 5 mg.

A-3.2 Calculation

$$\text{Moisture content, percent by mass} = \frac{100\,(M \times X)}{M}$$

where

M = mass, in g, of the material taken for test; and

X = mass, in g, of the material after drying.

A-4 DETERMINATION OF LEAD

A-4.1 Reagents

A-4.1.1 *Dilute Sulphuric Acid* — Approximately 2 percent (*v/v*).

A-4.1.2 *Ammonium Sulphate Solution* — 2 percent (*m/v*).

A-4.1.3 *Ammonium Acetate Solution (Neutral)* — 40 percent (*m/v*).

A-4.1.4 *Potassium Dichromate Solution* — 10 percent (*m/v*).

A-4.1.5 *Concentrated Nitric Acid* — *See* IS 264 : 1976.

A-4.2 Procedure

(Applicable for electrolytic manganese dioxide for orienting check on lead content).

A-4.2.1 10.0 g of manganese dioxide is weighed into a 1 000 ml upright beaker and dissolved with concentrated hydrochloric acid while being heated on hot plate. Then the solution is boiled down until almost dry. About 30 ml concentrated sulphuric acid is carefully added and the beaker contents evaporated on a hot plate to remove hydrochloric acid, continuing until thick white sulphuric acid fumes appear.

A-4.2.2 The mixture is allowed to cool, after which about 100 ml water is added carefully while the beaker is shaken. After boiling briefly the lead sulphate thus formed is allowed to precipitate for 2 to 3 hours. The sediment is flushed into hard filter with cold 2 percent sulphuric acid and washed briefly with cold 2 percent ammonium sulphate solution (do not use plain water).

A-4.2.3 The filter paper is now torn into two halves and each piece covered in a conical flask with about 50 ml of 40 percent ammonium acetate solution; the filter paper is reduced further still with a glass rod, heating at the same time, after about 5 minutes constant heating, the filter remnants are filtered off through a medium-hard filter. The flask is rinsed out with water only after the filter paper has drained. The filter paper is then washed thoroughly with hot water until there is about 400 ml of filtrate. This is diluted to about 500 ml, mixed with 15 to 30 ml concentrated acetic acid, brought to the boil and mixed with 5 ml ammonium acetate solution brought to the boil and mixed with 15 to 30 ml of hot 10 percent potassium chromate solution. This causes lead chromate to precipitate.

A-4.2.4 The solution above the precipitate shall be orange red, otherwise more potassium chromate solution shall be added. Boil for 15 to 30 minutes until the precipitate has agglomerated well, then the sediment is allowed to stand cold for 4 hours, after which it is filtered off on a pre-dried suction filter 1 G 4 whereby the liquid above the precipitation is poured through the suction filter, the sediment then swirled up with about 100 ml lukewarm water and poured into the filter flask. The beaker is rinsed out with lukewarm water (any lead chromate adhering to the beaker is removed with a rubber wiper) and the suction filter flushed two or three times with lukewarm water. The filter is dried at a temperature of 105°C until constant mass is reached (2 to 3 hours).

A-4.3 Procedure

(For manganese dioxide containing alkaline earths silicilic acid, lead sulphate, as well as soluble sulphate.)

A-4.3.1 10.0 g of manganese dioxide are weighed into 1 000 ml upright beaker and dissolved in concentrated hydrochloric acid while being heated on an electric hot plate. Then the solution is evaporated to the point of dryness, moistened with a little concentrated hydrochloric acid once or twice to render the silicilic acid insoluble and again evaporated to dryness. Add about 10 ml concentrated hydrochloric acid. The insoluble residue is filtered off through a tared filter and washed well with hot 10 percent acetic acid and water. Hot ammonium acetate solution is poured over the filtrate several times in order to prevent loss of lead as lead sulphate when separating the hydrochloric acid insolubles. The filtrate is then brought to a *p*H value of 2 with semi-concentrated hydrochloric acid or ammonium acetate solution (drop on to special indicator paper, *p*H range 0.5 to 5). The mixture is now heated. Pass hydrogen sulphide gas until it is cold. The resulting sulphide is filtered off and the paper completely incinerated wet in a 500 ml narrow necked conical flask with about 40 ml of a mixture (made up of 1 part by volume of concentrated sulphuric acid and 3 parts by volume of concentrated nitric acid), heating first until heavy white sulphuric acid fumes develop. The resulting dark brown colouration is removed by one or two additions of a few ml of the acid mixture on the rim of the flask and reheating until pure white fumes develop.

A-4.3.2 The mixture is allowed to cool, after which about 100 ml water is added carefully while the beaker is shaken. After boiling briefly, the lead sulphate thus formed is allowed to precipitate for 2 to 3 hours. The sediment is flushed into a hard filter with cold 2 percent sulphuric acid and washed briefly with cold 2 percent ammonium sulphate solution (on no account use pure water).

A-4.3.3 The filter paper is now torn into two halves and each piece covered in a conical flask with about 50 ml of 40 percent ammonium acetate solution; the filter is reduced further still with a glass rod, heating at the same time. After about 5 minutes constant heating, the filter remnants are filtered off through a medium-hard filter. The flask is rinsed out with water only after the filter has drained. The filter is then washed thoroughly with hot water until there is about 400 ml of filtrate. This is diluted to about 500 ml, mixed with 5 ml concentrated acetic acid, brought to the boil and mixed and with 25 to 30 ml of hot 10 percent potassium chromate solution. This causes lead chromate to precipitate.

A-4.3.4 The solution above the precipitate shall be orange red, otherwise more potassium chromate solution shall be added. Boil for 15 to 30 minutes until the precipitation has agglomerated well, then the sediment is allowed to stand cold for 4 hours, after which it is filter off on a predried suction filter. The sediment then swirled up with about 100 ml lukewarm water and poured into the filter flask. The beaker is rinsed out with lukewarm water (any lead chromate adhering to the beaker is removed with a rubber wiper) and the suction filter flushed two to three times with lukewarm water. The filter is dried at a temperature of 105°C until constant mass is reached (2 to 3 hours).

A-4.4 Calculation

$$\text{Lead (as Pb), percent by mass} = \frac{A \times 0.641\,0 \times 100}{M}$$

where

A = mass, in g, of the precipitate; and
M = mass, in g, of the material taken for the test.

A-5 DETERMINATION OF IRON CONTENT

A-5.1 Reagents

A-5.1.1 *Potassium Iodide Solution* — 10 percent aqueous (*m/v*).

A-5.1.2 *Starch Iodide Solution* — 1 percent (*m/v*). Make up fresh as required. Boil until completely clear.

A-5.1.3 *Standard Sodium Thiosulphate Solution* — 0.1 N.

A-5.1.4 *Standard Iron Solution* — 1 mg Fe^{+3} per ml.

A-5.1.5 *Concentrated Hydrochloric Acid* — *See* IS 265 : 1993.

A-5.2 Procedure

A-5.2.1 Weigh 10 g of artificial manganese dioxide into a wide-necked conical flask and dissolve in 50 ml concentrated hydrochloric acid. Reduce solution to syrupy consistency. Boil away chloride completely.

A-5.2.2 Add 50 ml water to the syrupy solution and pour in 10 ml potassium iodide solution. Shake the flask well, close with a rubber stopper and stand in a dark place for 10 minutes. Then add 1 ml starch solution, titrate rapidly with 0.1 N sodium thiosulphate solution until colourless or pale pink end point appears.

A-5.2.3 Where iron content is low, titrate with 0.01 N sodium thiosulphate solution. The normality of the 0.01 N thiosulphate solution shall be checked. For this purpose dilute 10 ml Fe^{+3} (solution) with 2 ml concentrated hydrochloric acid, about 80 ml water and 10 ml potassium iodide solution; titrate as defined above. The theoretical consumption is 17.9 ml of 0.01 N thiosulphate solution.

A-5.3 Calculation

For titration with 0.1 N sodium thiosulphate solution

$$\text{Iron (as Fe), percent by mass} = \frac{A \times N \times 0.559 \times N}{M}$$

For titration with 0.01 N sodium thiosulphate solution

$$\text{Iron (as Fe), percent by mass} = \frac{A \times N \times 0.055\,9}{M}$$

where

A = volume of thiosulphate solution consumed in respective cases;
M = mass, in g, of the material taken for test; and
N = normality factor of thiosulphate solution.

A-6 DETERMINATION OF SULPHATE

A-6.1 Reagents

A-6.1.1 *Concentrated Hydrochloric Acid* — *See* IS 265 : 1993.

A-6.1.2 *Barium Chloride Solution* — Approximately 10 percent (*m/v*).

A-6.2 Procedure

10 g of manganese dioxide is dissolved in about 100 ml concentrated hydrochloric acid. After the chlorine has been fully boiled away, the clear solution is concentrated until drying occur at the edge. Extract with dilute hydrochloric acid and filter off hydrochloric acid-insoluble matter.

The sulphate in the filtrate is precipitated as barium sulphate in the usual manner with barium chloride solution at boiling heat; transfer to the filter paper, wash with water as usual, reduce to ashes in a platinum or porcelain crucible and then weigh.

A-6.3 Calculation

Sulphate (as SO4), percent by mass = $M \times 4.115$

where

M = mass, in g, of the precipitate.

A-7 MATTER INSOLUBLE IN HYDROCHLORIC ACID

A-7.1 Reagents

A-7.1.1 *Concentrated Hydrochloric Acid* — *See* IS 265 : 1993.

A-7.2 Procedure

Weigh 5 g sample accurately and transfer into 500 ml tall conical beaker. Add 40-50 ml of concentrated hydrochloric acid and heat slowly under fume hood until all MnO_2 is decomposed. Boil off Hydrochloric Acid till mass becomes nearly syrupy. Add about 50 ml of distilled water rinsing the sides of beaker and 10 ml of Hydrchloric Acid and boil for 5-10 minutes.

Filter through Whatman filter paper No. 40, wash to make the residue free from soluble salts and acid, dry and ignite to burn off all organic matter. Weigh the residue.

A-7.3 Calculation

$$\text{Matter insoluble in acid, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass, in g, of the residue; and

M = mass, in g, of the sample.

A-8 DETERMINATION OF COPPER

(Not applicable for very low Cu as in EMD.)

A-8.1 Reagents

A-8.1.1 *Dithizone Solution*

15 mg dithizone is dissolved in 250 ml carbon tetrachloride and agitated mechanically. Watch for complete dissolution. Store in a dark place in tightly stoppered bottle. Always use the same 250 ml measuring flask, which must be thoroughly cleaned and dried.

A-8.1.2 *Hydrochloric Acid* — 0.5 N, 10 ml concentrated hydrochloric acid are diluted with water to make 2 000 ml.

A-8.1.3 *Standard Copper Solution* — Prepare same as in **A-15.1.6**.

A-8.1.4 *Hydrazine Sulphate Solution*

5 g hydrazine sulphate is covered with water in a 100-ml measuring flask; then 4 ml concentrated ammonia solution are pipetted in the flask filled to the mark with water and the mixture agitated. Keep in well closed bottle.

A-8.1.5 *Determining Standard of Dithizone Solution*

Immediately after preparation and every 14 days thereafter, the dithizone solution must be checked with cuprous salt solution (**A-8.1.3**). For this purpose, 1.85 ml of the cuprous salt solution are put in a graduated cylinder; the solution bottle is rinsed with a few drops of 0.05 N hydrochloric acid, without making up to 20 ml. When titrating with dithizone as per **A-8.2.2** reagent consumption should be 5 ml. The titer (*t*) found need be taken into account only if it is lower than 0.98 or higher than 1.02. It must not be less than 0.90 or more than 1.10.

A-8.2 Procedure

NOTE — Before use, all glassware must be left to stand or be shaken for 1 minute with semi-concentrated nitric acid. Rinse thoroughly with water.

A-8.2.1 *Preparation of Analytical Solution*

Weigh 0.20 g of manganese dioxide into a 150-ml beaker (use rapid balance), pour on 10 ml concentrated hydrochloric acid and then evaporate carefully just to the point of dryness. The residue is absorbed in about 30 ml of 0.05 N hydrochloric acid (**A-8.2**) and the solution boiled briefly (if Fe is present, the solution should turn yellowish). Persistent white turbidity does not matter. The solution is decanted into a 100-ml measuring flask together with 0.05 N hydrochloric acid, without filtration, and is filled to the graduation mark, with 0.05 N hydrochloric acid (**A-8.2**). Agitate vigorously. In order to determine the copper content, it is best to take a sample after settling of hydrochloric acid-insoluble matter.

A-8.2.2 Up to 20 ml of the analytical solution (depending on copper content) are pipetted into a small graduated cylinder; where the sample is less than 20 ml, make up to 20 ml with 0.05 N hydrochloric acid (A-8.2). Then 2 ml hydrazine sulphate solution (A-8.1.4) are added with the pipette and after rotating the cylinder the *p*H value is determined by dipping into Merck special indicator paper (ranger; *p*H 0.5 to 5). The *p*H value should be 1.5 to not more than 2.0. Add small amounts of dithizone solution (A-8.1.1) with a micro-burette, starting with portion of at least 0.1 ml, shaking after each addition until the colour has shifted to purple or greyish-violet. Towards the end of titration, agitate mechanically for 2 minutes after each addition of reagent. Titration is finished when a clear gray is obtained. Check by continuing to agitate for another minute; the colour should not change any more. Should the colour be difficult to discern, dilute with a few ml of carbon tetrachloride but do not titrate such diluted solution any further.

A-8.2.3 Where copper contents are very small it is necessary to agitate longer, for example, up to 5 minutes for 0.01 to 0.15 percent copper, and about 10 minutes for 0.005 to 0.01 percent (use automatic agitator).

A-8.2.4 A blank value is determined only if there is doubt that the reagents used contain Copper.

A-8.3 Calculation

$$\text{Copper (as Cu), percent by mass} = \frac{V \times 0.185 \times t}{V_1}$$

where

V = volume, in ml, of dithizone solution consumed;

V_1 = volume, in ml, of analytical solution; and

t = titre value of dithizone solution (A-8.1.5).

NOTE — Dithizone is a very sensitive reagent which decomposes easily, especially in the presence of alkali and in full light. It can react with nearly all metals, for which reason cleanliness must be particularly observed.

Since only very small amounts are proved or determined, the slightest impurities can lead to considerable errors. Therefore, all glassware must be cleaned before use (*see* Note under A-8.2). Owing to easy decomposition of dithizone the content must be determined with fresh solutions and checked occasionally during storage. The solution may lose at the most 1 percent of effective dithizone per week, otherwise the carbon tetrachloride used is unsuitable, or mistakes have been made in preparing the solution, or storage if at fault. Given perfect chemicals and proper procedure, decomposition will be around 0.1 percent only.

In the *p*H range of 1.5 to 2.0 the dithizonate of Bi (red-orange) is also extracted, as well as that of Ag (golden-yellow), of Au (golden-yellow) and of Hg (orange-yellow).

Where the Fe content is extremely high, it may be necessary to greatly reduce the sample of analytical solution in spite of hydrazine addition, in order to prevent oxidation of the dithizones induced by atmospheric oxygen. The lower reaction limit is thereby raised correspondingly; since it is not possible to work with less than 0.10 ml of dithizone solution.

Very useful is a 10 ml micro-burette with receptacle, an aerating stopper, and a super-tight 'Bistabil' flat cock.

A-9 DETERMINATION OF NICKEL

A-9.1 Apparatus

A-9.2 Reagents

A-9.2.1 *Citrate Tartrate Solution*

100 g of sodium citrate and 25 g sodium tartrate are dissolved in water and then made up to 500 ml.

A-9.2.2 *Dimethylglyoxime Sodium Salt*

1 percent aqueous solution or 1 percent solution of dimethylglyoxime in methyl alcohol.

A-9.2.3 *Hydroxylamine-Hydrochloride*

A-9.2.4 *Chloroform*

A-9.2.5 *Vaseline White*

A-9.3 Procedure

A-9.3.1 1 g of manganese dioxide is dissolved in a few ml of concentrated hydrochloric acid. Evaporate to one-half the volume, dilute with about 50 ml water, filter into a 250 ml separating funnel and then bring the filter up to a total volume of about 100 ml. In the following sequence, add 10 ml citrate-tartrate solution, a spatula-tip (20-25 mg) hydroxylamine-hydrochloride and ammonia (slowly) until a *p*H-value of 8 is reached (drop on to special indicator paper, range: *p*H 5.5 to 9). Finally, add 3 ml dimethylglyoxime solution. The mixture is stirred each time a reagent is added. Extract with 25 ml chloroform shaking for 30 seconds and allowing the extract to pass through a small, dry filter into a 50-ml measuring flask after phase separation. The filter is returned to the separating funnel and the contents again swirled out with 25 ml chloroform. The chloroform phase is filtered into the first extract through a fresh filter. The contents of the flask are made up to 50 ml. Photometric evaluation is done at 360 to 380 nm, an incandescent bulb with the S 38 filter being used in conjunction with the photometer.

The solution must be carefully protected against evaporation by covering the cuvettes immediately with grooved covers. Chloroform serves as reference solution. Fresh reagents are to be checked for absence of nickel by means of blank test. Should a blank value occur, it must be deducted from the measured value.

A-9.4 Calculation

$$\text{Nickel, in mg} = \frac{\text{measured extinction}}{\text{deposite length in cm}} \times 0.895$$

With the weighed samples referred to above, the nickel content is determined as follows:

$$\text{Nickel (as Ni), percent by mass} = \frac{\text{measured extinction}}{\text{deposite length in cm}} \times 0.895$$

If other photometers and filters are used, a calibration curve must be established and the corresponding factor computed on a straight line.

NOTE — Chloroform extracts can be collected for the purpose of recovering the solvent. One slow distillation in a column of about 30 cm length will suffice. The first few ml are discarded and the distillate grading between 60 and 61 is collected in pure ethyl alcohol amounting to percent of the total volume.

Where Ni content is more than 0.12 percent, evaluation must be repeated with a correspondingly smaller quantity of manganese dioxide. Above this limit the photometric evaluation is not exact and furthermore the solubility of Ni dimethylglyoxime in chloroform is very limited (1.7 mg Ni in 50 ml $CHCl_3$).

A-10 DETERMINATION OF PARTICLE SIZE (WET METHOD)

A-10.1 Apparatus

A-10.1.1 *IS Sieve* — 75 microns.

A-10.2 Reagents

A-10.2.1 *Wetting Agent* — like teepol, laboleen, etc.

A-10.3 Procedure

Weigh 50 g of the sample into a 250 ml beaker. Add about 100 ml of water and few drops of wetting agent. Mix thoroughly with the help of a glass rod for proper wetting. Transfer this slurry into a 75 micron IS sieve placed in the laboratory sink with the help of a water jet. Keep on washing the residue on the sieve with water till all the finer particles passes through the sieve. If required, add few more drops of wetting agent on the sieve and continue washing with water into the sieve till the water passing through the sieve is free from any particles. Dry the residual material on the sieve. Cool and weigh it accurately.

A-10.4 Calculation

$$\text{Particle size, percent by mass} = \frac{M - M_1}{M} \times 100$$

where

M = mass in g of sample, and

M_1 = mass in g of residue.

A-11 DETERMINATION OF *p*H VALUE

A-11.1 Procedure

Weigh 10 g sample and transfer into 250 ml conical flask. Add 200 ml distilled water and put rubber stopper. Shake on wrist action shaker or any similar device for one hour. Transfer the contents into a 250 ml beaker and allow to settle for about 30 minutes. Measure *p*H value of supernetent solution using *p*H meter. (slight black turbidity in supernetent solution does not materially affect the results). The *p*H meter should have been previously calibrated using standardized buffer solutions.

A-12 DETERMINATION OF CRYSTAL STRUCTURE

A-12.1 Apparatus

X-ray diffraction unit with iron target tube.

A-12.2 Procedure

Find out the crystal structure by powder X-ray diffraction technique using camera or diffractometer to find out *d* spacing values. Iron K-alfa radiation is used.

Gamma MnO_2 is characterized by *d* spacings corresponding to Cu the following:

	d Spacing in A^0
1	3.92-4.0
2	2.4
3	2.12
4	1.63
5	1.39

On diffractogram, a broad hump will be obtained corresponding to the first *d* spacing, indicating amorphous nature of product. The intensity (height) of this peak may vary from moderate to low and sometimes so low as to be barely discernible. Peaks corresponding to the rest of the spacings are sharp.

Alpha and Beta type of MnO_2 samples show peaks corresponding to 6.96 and 3.11 A° *d* spacings besides other peaks which are common to all except at 3.92-4.

NOTE — Considerable experience is required to make judgement of crystal structure by X-ray diffraction methods.

A-13 ALTERNATE METHOD FOR DETERMINATION OF IRON

A-13.1 Reagents

A-13.1.1 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

A-13.1.2 *Hydroxylamine Hydrochloride Solution* — 10 percent (*m/v*).

A-13.1.3 *Ammonium Acetate Solution* — 25 percent (*m/v*).

A-13.1.4 *Ortho-phenanthroline Solution* — 0.1 percent (*m/v*).

A-13.1.5 *Standard Iron Solution*

Dissolve 7.02 g of ferrous ammonium sulphate [$FeSO_4 (NH_4)_2SO_4.6H_2O$] in the mixture of 2 ml of hydrochloric acid (1 : 1) and water and transfer to a 1 000 ml volumetric flask and dilute with water to the mark.

Pipette 10 ml of this solution into another 1 000 ml volumetric flask and dilute with water to the mark. One ml of this solution contains 0.01 mg of iron.

A-13.2 Apparatus

A-13.2.1 *Spectrophotometer* — UV type.

A-13.3 Procedure

Dilute 5 ml of 'A' solution as prepared in **A-5.2.1** (containing 0.50 g of the sample) with water to 50 ml and take 10 ml (containing 0.10 g of the sample) into a beaker. Add 1 ml of hydrochloric acid (1 : 1), 1 ml of hydroxylamine hydrochloride solution, 5 ml of ammonium acetate solution and 2 ml of *o*-phenanthroline solution and dilute with water to 25 ml to make *p*H of the solution to about 4.5. After standing for 20 minutes to develop, colour, take the solution into the photometric cell, and measure the absorbency at the wavelength of approximately 510 nm. Correct the absorbency at the wavelength of approximately 510 nm. Correct the value by carrying out the blank test and find the iron content from the calibration curve (**A-13.3.1**).

A-13.3.1 *Preparation of Calibration Curve*

Take several aliquots of iron standard solution varying from 0 to 7 ml and proceed as described above beginning the addition of the reagents and prepare the calibration curve in relation to the iron content and absorbency.

A-13.4 Calculation

Iron (as Fe),

$$\text{percent by mass} = \frac{\text{mg Fe (obtained from curve)}}{\text{sample mass taken for test}} \times 100$$

A-14 ALTERNATE METHOD FOR DETERMINATION OF LEAD

A-14.1 Reagents

A-14.1.1 *Hydroxylamine Hydrochloride Solution* — 10 percent (*m/v*).

A-14.1.2 *Gelatin Solution* — 0.1 percent (*m/v*).

A-14.1.3 *Standard Lead Solution*

Dissolve 1.60 g of lead nitrate in water. Transfer the solution to a 1 000 ml volumetric flask and dilute with water to the mark. 1 ml of this solution contains 1 mg of lead.

A-14.2 Apparatus

A-14.2.1 *Polarograph*

A-14.3 Procedure

Take 20 ml of 'A' solution as prepared in **A-5.2.1** (containing 2.0 m of the sample), add 1 mg of hydroxylamine hydrochloride solution and warm to reduce iron. After cooling, add 5 ml of gelatin solution, dilute with water to 50 ml and stir the solution thoroughly. Take suitable amount of the solution into an electrolytic vessel and pass in nitrogen or hydrogen gas for about 10 minutes to remove dissolved oxygen.

A-14.3.1 Then plot the polarogram between the potential −0.2 to −0.7 V. Measure the wave height produced at the half-wave potential (approximately) at −0.4 V *vs* standard calomel electrode and find the lead content from the calibration curve (**A-14.3.2**) previously prepared.

A-14.3.2 *Preparation of Calibration Curve*

Take several aliquots of lead standard solution varying from 0 to 3 ml. Proceeding as described above, draw the curve in relation to the lead content and wave height and then draw a parallel line from the zero point to prepare the calibration curve.

A-14.4 Calculation

Lead (as Pb),

$$\text{percent by mass} = \frac{\text{mg of Pb (obtain from curve)}}{\text{sample mass taken for test}}$$

A-15 ALTERNATE METHOD FOR DETERMINATION OF COPPER

A-15.1 Reagents

A-15.1.1 *Hydroxylamine Hydrochloride Solution* — 10 percent (*m/v*).

A-15.1.2 *Citric Acid Solution* — 10 percent (*m/v*).

A-15.1.3 *Dilute Ammonium Hydroxide* — 1 : 3 (*v/v*).

A-15.1.4 *Neocuprion Solution* — 0.1 percent in ethyl alcohol (*m/v*).

A-15.1.5 *Chloroform*

A-15.1.6 *Standard Copper Solution*

Dissolve 3.93 g of copper sulphate ($CuSO_4.5H_2O$) in water and transfer to a 1 000 ml volumetric flask and dilute with water to the mark. Pipette 10 ml of this solution into another 1 000 ml volumetric flask and dilute with water to the mark. 1 ml of this solution contains 0.01 mg of copper.

A-15.2 Apparatus

A-15.2.1 *Spectrophotometer* — UV type.

A-15.3 Procedure

Take 20 ml solution as prepared in A-5.2.1 (containing 2.0 g of the sample) into a 200 ml beaker and dilute with water to 50 ml. Add 5 ml of hydroxylamine hydrochloride solution and 5 ml of citric acid solution followed by addition of 10 to 12 ml dilute ammonium hydroxide and transfer of the solution to 100 ml separating funnel, add 2 ml of neocuprion solution and shake the funnel vigorously. Then add exactly 10 ml of chloroform, shake the funnel vigorously for 2 minutes and allow to stand the solution to separate into the aqueous and chloroform layers. Transfer the chloroform layer to the cell of the photometric cell and measure the absorbency of the solution at the wavelength of approximately 454 nm. Correct the value by carrying out the blank test and find the copper content from the calibration curve (A-15.3.1).

NOTE — If the separation of the solution is incomplete, transfer the chloroform layer in a 50 ml beaker, add about 1 g of anhydrous sodium sulphate and stir well with a glass rod to dehydrate the solution.

A-15.3.1 *Preparation of Calibration Curve*

Take several aliquots of copper standard solution varying from 0 to 6 ml and proceed as described above, beginning the addition of the reagents and prepare the calibration curve in relation to the copper content and absorbency.

A-15.4 Calculation

Copper (as Cu), percent by mass

$$= \frac{\text{mg Cu (obtained from curve)}}{\text{sample mass taken for test}} \times 100$$

A-16 DETERMINATION OF LEAD, IRON, COPPER, AND NICKEL BY ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHODS (REFERENCE METHODS)

A-16.1 Reagents

Any good make Atomic Absorption Spectrophotometer with A-Acetylene flame and having background correction facilities can be used.

A-16.2 Reagents

1. *Concentrated Hydrochloric Acid* — A.R. grade.
2. Standard aquious metal salt solutions containing :

Lead	—	2, 4, 8, 18	mg Pb/litre
Iron	—	1, 2, 4	mg Fe/litre
Copper	—	0.25, 0.5, 1	mg Cu/litre
Nickel	—	1, 2, 4	mg Ni/litre

The above standard solutions are prepared by dissolving pure salts or metals in hydrochloric acid or nitric acid. The final diluted solution should contain about 1 percent HCl for ensuring good keeping qualities.

A-16.3 Procedure

Weigh 4 g of sample accurately and transfer in a beaker. Add about 50 ml hydrochloric acid and heat gently first and later on bring to boiling. When all MnO2 particles are decomposed, add about 20 ml of distilled water and boil for few minutes. Cool and transfer the contents in 100 ml volumetric flask. Rinse the beaker, wash and transfer the same to the volumetric flask and dilute to 100 ml with distilled water. After mixing, filter the solution if required to remove any insolubles.

This is a solution A.

Pipette out of 10 ml of solution A and dilute to 100 ml in a volumetric flask with distilled water. This is a solution B.

Solution A is used for determination of lead, copper and nickel and solution B is used for determination of iron.

Atomic Absorption Spectrophotometer is switched on and using hollow cathode lamps of respective elements (Pb, Cu, Ni, and Fe) the instrument is adjusted for various operating conditions as per the instrument manual. Slightly oxidizing flame is maintained for analysis. Monochromator is adjusted at following wavelengths for different elements while analysing for the same:

Element	*Wavelength*
Lead	217.0 nm
Copper	324.7 nm
Nickel	232.0 nm
Iron	248.3 nm

Typically different standard solutions are aspirated in the flame and absorbance values are recorded followed by that using sample solution. Concentration of the element under question is found out by comparison with standard from the above data using rule of three, from graphs or by aid of microprocessor.

Generally for the above standard solutions, a straight line curve is obtained when concentration is plotted against absorbance values. In most of the modern instruments a provision for automatic background correction is provided and it should be used for determination of the above elements.

A good experience and familiarity conforming the instrument is essential for a person carrying out analysis.

A-16.4 Calculation

Lead (as Pb), percent by mass =

$$\frac{\text{mg/lit of Pb (Obtained in solution A from curve)}}{\text{Sample mass taken for test} \times 100}$$

Copper (as Cu), percent by mass =

$$\frac{\text{mg/lit of Cu (Obtained in solution A from curve)}}{\text{Sample mass taken for test} \times 100}$$

Nickel (as Ni), percent by mass =

$$\frac{\text{mg/lit of Ni (Obtained in solution A from curve)}}{\text{Sample mass taken for test} \times 100}$$

Iron (as Fe), percent by mass =

$$\frac{\text{mg/lit of Fe (Obtained in solution B from curve)}}{\text{Sample mass taken for test} \times 100}$$

ANNEX B

(*Clause* 6)

SAMPLING OF MANGANESE DIOXIDE FOR DRY BATTERIES

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test sample, the following precautions and directions shall be observed.

B-1.1 Sample shall not be drawn in an exposed place.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled and the sampling instrument from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.4 The sampled material shall be placed in clean, dry, air-tight glass or other suitable containers which have no action on the material.

B-1.5 Each sample container shall be sealed air-tight with a stopper after filling and shall be marked with all particulars of the material and the date of sampling.

B-2 SCALE OF SAMPLING

B-2.1 Lot

In a single consignment of the material all the containers of the same type and the size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different types and sizes of containers, the containers belonging to the same batch, type and size shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

Table 2 Scale of Sampling

Number of Containers in the Lot (*N*)	Sample Size (*n*)
(1)	(2)
Up to 50	3
51 " 100	4
101 " 150	5
151 " 300	7
301 " 500	10
501 and above	15

B-2.1.2 These containers shall be selected at random from the lot. In order to ensure randomness of selection, random number tables (*see* IS 4905 : 1968) shall be used. In case such tables are not available, the following procedure may be used:

Arrange the containers in the lot in a systematic order and starting from any one count them as 1, 2, 3,............, etc, up to *r* and so on where *r* is the integral part of *N*/*n* (*N* and n being the lot size and sample size respectively). Every rth container thus counted shall be withdrawn to constitute the sample.

B-3 PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected, draw a small representative portion of the material approximately 250 g in mass.

B-3.2 Out of these portions, small but equal quantity of the material shall be taken and mixed thoroughly to form a composite sample of mass about 150 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier, and the third to be used as a referee sample.

B-3.3 From the remaining portions of the material from each container, draw about 200 g of the material. Divide it into three equal parts and transfer the material to three separate bottles giving full identification particulars of the sample on the bottles. The material in each bottle constitutes an individual sample. One of these three sets (each set containing a bottle representing *n* containers sampled) shall be for the purchaser, one for the supplier and the third for the referee.

B-3.4 The referee samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier and shall be used in case of dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of moisture content and manganese dioxide content shall be performed on each of the individual sample.

B-4.2 Tests for the determination of all the remaining characteristics given in Table 1 shall be carried out on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Sample

From each set of individual test results moisture content and manganese dioxide content, respective average ($\overline{X}$) and the range (R) of test results shall be computed (range R being defined as the difference between the maximum and the minimum values of test results) and the conditions specified in col 6 of Table 3 shall be satisfied.

B-5.2 For Composite Samples

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test results shall meet the corresponding values specified in the table of requirements.

Table 3 Criteria for Conformity for Moisture and Manganese Dioxide Content
(*Clause* B-5.1)

Sl No.	Characteristic	Test Result	Average	Range	Criterion for Conformity
(1)	(2)	(3)	(4)	(5)	(6)
i)	Moisture content	1,2,3,....n	$\overline{X}_1$	R_1	$\overline{X}_1 + 0.6\ R1$ shall be less than or equal to the prescribed limit in Table 1
ii)	Manganese dioxide	1,2,3,....n	$\overline{X}_2$	R_2	$X_2 - 0.6\ R_2$ shall be greater than or equal to the prescribed limit in Table 1

ANNEX C

(*Foreword*)

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This Indian Standard has been developed from Doc : No. CHD 003 (0392).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Simco Printing Press, Delhi